## News Briefs

#### **General Developments**

Inquiries about News Briefs, where no contact person is identified, should be referred to the Managing Editor, Journal of Research, National Institute of Standards and Technology, Building 101, Room E215, Gaithersburg, MD 20899-2500; telephone: (301) 975-3577.

## NIST UNCOVERS POTENTIAL PROBLEM FOR SEMICONDUCTOR LITHOGRAPHY

NIST researchers have uncovered a potentially serious optical problem affecting designs for future generations of semiconductor manufacturing equipment using deep ultraviolet light. The "Moore's Law" phenomenon the doubling of chip complexity with each generation—has been possible largely because of continual advances in lithography, allowing manufacturers to image and process integrated circuits with smaller and smaller dimensions. Reaching very small dimensions requires using very short-wavelength light. Current state-of-theart production processes use deep ultraviolet lasers at 248 nm and 193 nm to image circuits with critical features as small as 130 nm to 150 nm. The next major steps for lithography are expected to be systems using 157 nm light, ultimately achieving feature sizes in the range of 70 nm.

Some of the optics for 193 nm and all of the optics for 157 nm lithography are made of calcium fluoride, one of only a few materials that are transparent at 157 nm. A NIST physicist recently showed that calcium fluoride is inherently birefringent in the deep ultraviolet—meaning that the crystal refracts light differently depending on the polarization of the light. Other NIST physicists confirmed these results theoretically.

The practical import for designers is that a calcium fluoride lens will not focus properly without, at the very least, careful control of the light as it enters the lens and a design that accounts for this intrinsic birefringence. This is a difficult problem given that there are about 20 such lens elements in a typical stepper or optical lithography lens. The modeling software used to design such

systems is being modified to account for the effects of birefringenc, but it is not yet ready.

NIST researchers are pursuing potential solutions to the problem involving mixed crystals to compensate for the birefringence effect. Details of the findings were first made public at the International SEMATECH-sponsored International Symposium on 157 nm Lithography.

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## NIST SMOOTHS TRANSITION TO "SAFE," ENERGY-EFFICIENT REFRIGERATION

The National Institute of Standards and Technology has contributed significantly to the refrigeration industry through its work with "alternative" refrigerants for the past 14 years, and the effort is paying off.

When it became known that chlorofluorocarbons and hydrochlorofluorocarbons in refrigerants were contributing to depletion of the earth's protective ozone layer, NIST began a program (funded by the Department of Energy's Office of Basic Energy Sciences and Office of Building Technologies) to measure the properties of their replacements. These data have been made available through a computer program known as NIST Standard Reference Database 23: Thermodynamic and Transport Properties of Refrigerants and Refrigerant Mixtures Database (or REFPROP for REFrigerant PROPerties). Some 1500 copies of REFPROP have been distributed over the years and it continues to be a timely and valuable tool for refrigeration engineers, chemical and equipment manufacturers, and others who use refrigerants. Version 6.0 now provides data on 33 pure refrigerants, as well as refrigerant mixtures.

But the database's real impact has been in facilitating the design of more energy-efficient cooling equipment. In 1999, the Environmental Protection Agency estimated that new, non-CFC chillers used to cool large buildings reduced U.S. energy costs by \$480 million annually (which corresponds to an improvement in energy efficiency of 35 %).

"Accurate property data are essential for optimizing efficiency, and the entire NIST refrigerants program over its 14 year duration would be paid for by a single years savings resulting from a single percentage point gain in energy efficiency for this single class (non-CFC chillers) of refrigeration equipment," says a new technical paper from NIST.

For a copy of this paper (26-01), contact Sarabeth Harris, NIST, MC104, Boulder, CO 80305-3328; (303) 497-3237; sarabeth@boulder.nist.gov. The REFPROP database may be ordered via the World Wide Web at http://www.nist.gov/srd/nist23.htm.

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## NEW SECURITY STANDARD FOR FEDERAL AGENCIES EFFECTIVE IN NOVEMBER

Computer security experts at the National Institute of Standards and Technology have developed a new standard for information scrambling products used by civilian federal agencies. The standard, NIST Federal Information Processing Standard 140-2, Security Requirements for Cryptographic Modules, becomes effective November 25, 2001.

Computer security products used by agencies for sensitive, unclassified information must be certified under the new FIPS standard. It replaces a standard, 140-1, that had been in place since 1994.

Accredited private sector laboratories have tested and validated more than 150 cryptographic modules as conforming to the existing standard. Indeed, the list is a "who's who" of cryptographic and information technology vendors and developers from the United States, Canada, and abroad. The list contains a complete range of security levels and a broad spectrum of product types including secure radios, Internet browsers, VPN devices, PC postage equipment, cryptographic accelerators, and others.

Vendors who plan to sell security equipment to the federal government submit their products and systems to the Cryptographic Module Validation Program. The CMVP is a joint program between NIST and its Canadian counterpart, the Communications Security Establishment. Before the launch of the testing program in 1995, there was no generally accepted way to test cryptographic modules. The cryptographic modules may be any combination of hardware, software and firmware.

While the government agencies oversee the program, all of the nuts-and-bolts testing is done by private,

accredited laboratories in the United States and Canada. The program tests ensure that a product meets federal standards. Federal agencies are currently required to use FIPS 140-1 when purchasing cryptographic products intended to protect information. Additionally, the standards are used in the private sector as well, particularly in the financial services industry.

Detailed information about the new standard is available at http://www.nist.gov/fips140-2.

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#### BROCHURE SHOWS NIST RESEARCH, SERVICES BENEFIT DATA STORAGE EFFORTS

There's an unrelenting space crunch in magnetic data storage. By 2006, the storage industry aims to pack 0.16 Tbit/cm² on state-of-the-art magnetic disk drives—a recording density 40 times greater than todays top commercial offerings. The technical hurdles to be scaled are formidable.

The National Institute of Standards and Technology is helping industry to accomplish its ambitious goals. A new brochure describes key programs and resources that the institute is devoting to the cause. It profiles activities in 13 areas, including imaging and modeling of magnetic properties, nanometer-scale measurement capabilities and spintronics. Contacts are listed for each project or service. Also highlighted are specialized facilities such as NISTs Center for Neutron Research, an array of microscopes that provide finely detailed views of magnetic materials and an elaborately instrumented magnetic thin-film deposition laboratory.

NIST staff expertise spans all fields relevant to magnetic data storage, including materials science, electrical engineering, physics, mathematics and modeling, tribology, manufacturing engineering, chemistry, metrology and combinatorial methods. By illustrating the diverse range of research and resources NIST offers, the new brochure makes it easier for companies and their organizations working in the rapidly advancing technology area to take full advantage of NIST capabilities.

To obtain a copy of the Magnetic Data at NIST brochure, contact the NIST Inquiries Office at (301) 975-NIST (975-6478) or inquiries@nist.gov. Additional information on NISTs contributions to magnetic data storage may be found at http://www.nist.gov/computers. Media Contact: Mark Bello, (301) 975-3776; mark. bello@nist.gov.

#### NIST OFFERS ONLINE METROLOGY RESOURCE FOR ELECTRONICS MANUFACTURERS

Semiconductor, electronics and data storage device manufacturers can use a new NIST web page to easily find the NIST research, products and services of greatest relevance to their industry. The NIST semiconductor/ electronics industry-sector web page (www.nist.gov/public\_affairs/semiconductor.htm) is designed to help industrial R&D departments and manufacturing operations find the most accurate measurements, standards, calibrations and data available from NIST. Links to NIST cooperative research and funding opportunities also are offered.

The semiconductor/electronics web page gives very short descriptions of what NIST does to help build better microchips, from more accurately measuring step heights, dielectric films and interconnects to ways to improve manufacturing processes. Along with the brief project descriptions, the page offers links to more detailed descriptions of each project or program, as well as contact names, e-mail addresses and phone numbers.

The page is one of several new industry-sector web pages intended to improve industry awareness of NIST products, services and programs. Go to Information for Industry on the NIST home page (www.nist.gov) to access the index for all of the NIST industry-sector web pages.

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## NEW REFERENCE MATERIAL SIMPLIFIES SEM PERFORMANCE CHECKS

The use of a new "sharpness" reference material from NIST combined with data from state-of-the-art inspection software could help make routine the currently difficult but critical task of running performance checks on scanning electron microscopes, known as SEMs.

All SEMs, whether they are in the laboratory or on the production line, slowly lose performance ability with use. Loss in image quality also means loss in measurement sensitivity. Contributing to SEM performance loss are a variety of factors including misalignment, contamination and increase in size of the primary electron beam. Measuring the loss in image sharpness is one way to identify this performance.

An improved ability to assess SEM performance loss would be an important quality control advance for the more than \$200 billion semiconductor industry because fully automated SEMs are used to inspect silicon wafers.

NIST Reference Material 8091 is a small (approximately 2 mm²) diced semiconductor chip with tiny tower-shaped structures of silicon generated by a plasma-etching artifact commonly referred to as "grass." The fine-grained "towers" can be used to determine image sharpness at magnifications in excess of 100 000 times at both high- and low-accelerating voltages.

RM 8091 can be mounted onto a wafer, wafer piece or specimen stub for insertion into a laboratory SEM or wafer inspection SEM. The chip also can be mounted onto a "drop-in" wafer. It is designed for use with Fourier analysis software such as the NIST/SPECTEL SEM Monitor Program, the NIST Kurtosis program, the University of Tennessee SMART program, or similar analytical techniques.

The SEM Monitor is a collaborative effort by NIST, and industry. The SEM Monitor was honored in 1998 with an R&D 100 award from *Research and Development* Magazine. The system can make sharpness measurements on static, collected images or in real-time live mode, thus enabling users to easily adjust and align a CD-SEM or laboratory microscope to optimize performance.

RM 8091 is available from the NIST Standard Reference Materials Program. Purchases of RM 8091 can be made by calling (301) 975-6776. RM 8091 also can be ordered online at www.nist.gov/srm. For technical information, contact Michael Postek, (301) 975-2299.

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#### 24 000 VESTS PURCHASED BASED ON SPECIFICATION FOR STAB-RESISTANT PROTECTIVE GEAR

The specification developed and issued in September 2000 by NIST's Office of Law Enforcement Standards (OLES) for the National Institute of Justice (NIJ) for stab-resistant personal protective gear is now in use. For the first time, state, county, and local law-enforcement and correction agencies have been able to purchase NIJ-approved stab-resistant vests through funding from the Department of Justice (DOJ) Vest Partnership Program. NIJ is an agency of the DOJ and a principal sponsor of OLES.

According to the DOJ Office of Justice Programs, about 24 000 vests have already been purchased with some 17 000 going to correctional facilities. The NIJ approval process is based on specifications and associated tests developed by OLES. The internal construction of protective vests intended to absorb bullet impact

without permitting penetration has not proven to be effective protection from stabbing attack. Officers in correctional facilities in particular face the threat of blades made by inmates from any scrap of suitable metal available. In some countries, stabbing is a more prevalent threat in general society than bullets. The OLES stab-test fixture is a development of work carried out in the United Kingdom by the Police Scientific Development Branch. An article in the June issue of *Police Chief*, a publication of the International Association of Chiefs of Police, provided the information on the first purchase of stab-resistant vests through the DOJ Program to its wide audience of corrections and public safety officials.

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# DETERMINATION OF THE NON-UNIQUENESS OF THE INTERNATIONAL TEMPERATURE SCALE OF 1990

In the range -259 °C to 962 °C, temperatures on the International Temperature Scale of 1990 (ITS-90) are defined at a set of natural fixed-points that have assigned temperature values. Between these fixed points, standard platinum resistance thermometers (SPRTs) are used as interpolating devices with a prescribed interpolation formula. At any temperature between the fixed-point values, the temperature indicated by an SPRT may depend on the physical or chemical characteristics of that particular SPRT. This irreproducibility of temperature values is one type of so-called non-uniqueness of the ITS-90 and is an inherent limitation of the ITS-90. Although non-uniqueness has been quantified at lower temperatures, measurements at temperatures above 660 °C demand exceptional skill at achieving thermal equilibrium between multiple thermometers, performing accurate resistance measurements, and preventing chemical contamination of the thermometers over periods of many months. A recent paper by two NIST scientists culminates a multiyear effort on making these technically difficult measurements and is a major contribution to the metrology of the ITS-90.

These results were presented at the TEMPMEKO 2001 conference in Berlin and will be published in the conference proceedings. The NIST measurements in this range have uncertainties an order of magnitude smaller than all other published measurements, and set a conservative upper bound on the non-uniqueness of approximately 0.003 °C in the temperature range 660 °C to 962 °C. This bound is sufficiently low to demonstrate that the non-uniqueness of the ITS-90 in this temperature range may be neglected for all present industrial

temperature measurements. The reduction of uncertainty of the NIST dissemination of temperature scales enables secondary calibration laboratories and laboratories in the aerospace and electrical generation industries to perform traceable temperature measurements more readily at a given level of user uncertainty.

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## NIST DEVELOPS A NEW GENERATION OF FREQUENCY AND TIME STANDARDS

NIST staff members in Boulder, in collaboration with the Max Planck Institute in Germany, have demonstrated for the first time operation of an optical frequency standard with a microwave output. Their work signals the introduction of a new generation of atomic clocks with potential performance well beyond those in operation today. This first demonstration of an optical clock with a microwave output, reported in the August 3 issue of *Science*, involves an optical frequency standard coupled to a new optical-frequency synthesizer that bridges an octave in the optical spectrum and provides for a direct microwave output that is phase locked to the optical standard. The potential relative uncertainty of the optical clock is  $1 \times 10^{-18}$ , a factor of 1000 better than today's best standards.

Essential components of this standard include a single trapped mercury ion in a cryogenic trap, an ultra-stable laser used to probe the transition in the mercury ion, and a femtosecond laser system that produces a frequency comb spanning a full octave in frequency. In comparisons with a laser-cooled calcium optical standard, an upper limit for the fractional instability of  $7 \times 10^{-15}$  was measured in a 1 s averaging interval, which is significantly better than that of the worlds best microwave atomic clock.

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## NIST DEVELOPS PHASE-MODULATION SERVOS FOR ATOMIC CLOCKS

NIST scientists in Boulder, in collaboration with he Jet Propulsion Laboratory (JPL), have developed an improved modulation method for laser-cooled atomic clocks, providing for a high level of immunity to vibrations and substantial reduction of a number of systematic frequency shifts that can affect these clocks. The concept involves phase modulation of the interrogating microwave field, rather than the traditional frequency modulation used in most atomic clocks. In this new scheme, the phase of the microwave field in the first portion of the Ramsey cavity is fixed and the phase

in the second Ramsey region is varied relative to the phase in first region. The advantage of the method is that the frequency of the microwave field can be kept continu ously on the center of the resonance, rather than being stepped from one side of the resonance to the other, as is done using frequency modulation. At the peak of the resonance the clock is substantially less sensitive to vibration and to systematic effects than it is when the system resides most of the time on the steepest portion of the resonance curve.

The concept was initially developed to address the vibration sensitivity of the laser-cooled clock on the Primary Atomic Reference Clock in Space. In this collaborative program, involving NIST, JPL, the University of Colorado, and the Harvard-Smithsonian Center for Astrophysics, a laser-cooled cesium clock will be put aboard the International Space Station in 2005 to perform certain tests on gravitational theory and to improve upon the realization of the second. The modulation concept was tested on the NIST cesium-fountain clock, NIST-F1, and found to work so well that it has become the preferred mode of operation. It has also been picked up by others and is being used on other fountain clocks around the world.

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#### NIST DEVELOPS A NEW METHOD FOR PHASE AND AMPLITUDE NOISE MEASUREMENTS BETWEEN 10 GHZ AND 100 GHZ

NIST researchers in Boulder have developed a system for making high-resolution phase-and-amplitude-noise measurements to support the characterization of high-performance radars that use digital methods of signal processing. The new measurement system, which operates in the frequency range from 10 GHz to 100 GHz, uses the so-called two-channel cross-correlation method. The key to making these measurements stems from the generation of extremely stable reference signals which are synthesized by the combination of an ultra-stable sapphire resonator (oscillator), a set of low-noise regenerative dividers, and a set of cavity-stabilized Gunn oscillators. To date there have been no high-stability reference sources for measurements across this frequency region.

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## NEW TOOL FOR PLASMA ETCHING DIAGNOSTICS DEVELOPED BY NIST

The understanding and control of the complex chemical processes that occur in semiconductor plasma-etching

reactors is critical for the microelectronics industry, as it moves toward implementing multistep etching processes, to produce smaller and higher-aspect-ratio features. NIST scientists have developed a new spectroscopic technique for characterizing the complex chemistry that occurs in a semiconductor plasmaetching reactor. The technique uses linear absorption spectroscopy to determine the density and temperature of plasma species, such as radicals, ions, and molecules, along the radiation path. The technique represents a significant improvement over previous methods, such as infrared diode-laser spectroscopy, by providing unambiguous molecular identification over a broad range of molecular species, and absolute concentrations and temperatures. The instrumentation fits in two suitcases, making it convenient for setting up at a remote plasma reactor.

Initial tests near 0.1 THz demonstrated the detection of important fluorocarbon species in a triflouromethane plasma, such as might be used for etching silicon wafers. Present efforts are being made to extend the measurement frequency to near 1 THz.

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## NIST SCIENTISTS OBSERVE DYNAMICAL TUNNELING

The quantum mechanical phenomenon of tunneling, which allows a system to go from one state to another through a region that is classically forbidden, has been observed since the early days of quantum mechanics. Tunneling usually involves a particle going from one region to another through a barrier that classically the particle does not have sufficient energy to go over. Dynamical tunneling, predicted only since the early 1980s, is a similar phenomenon where some other constant of the motion other than energy forbids classically the quantum-mechanically allowed motion. Imagine a wheel spinning either clockwise or counterclockwise. Energetically, there is no difference between the two motions, but once you start the wheel spinning in one direction, there is no classical way to get it spinning in the other direction without stopping the wheel first. Quantum mechanically, however, this reversal may be accomplished without first stopping the

In an experiment performed by NIST, in collaboration with researchers from the University of Queensland, Australia, ultra-cold atoms oscillating in optical potential wells were observed to tunnel from one allowed motion to another which was not well connected via a classical pathway. Atoms initially loaded into the

bottom of the optical potentials were induced to oscillate back and forth. The number of atoms in a particular oscillatory motion was observed to decrease with time, as a group of atoms oscillating 180° out of phase with the initial motion began to appear. Eventually, almost all of the atoms end up in the out-of-phase motion but then tunneled back to the initial mode of oscillation. The researchers observed up to eight coherent transfers of atoms back and forth between the two stable motions due to dynamic tunneling, and no atoms were observed to exhibit intermediate motion between the two stable oscillatory motions.

The observation of dynamic tunneling in a well-characterized atomic system opens the door to future studies on the role of decoherence in quantum phenomena. Such studies are crucial for determining the role of decoherence in quantum information processing systems. The results of the experiment were published in the July 5th issue of *Nature*.

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#### IF YOU CAN DEFINE IT, WE CAN FIT IT

To expand the scope and improve usefulness to industry of the Algorithm Testing and Evaluation Program-Coordinate Measuring Systems (ATEP-CMS), a NIST scientist has been developing reference-fitting algorithms for arbitrarily complex rigid surfaces. The main fitting algorithm is now complete and undergoing testing. The algorithm requires the definition of a surface and a means to find the closest point on a surface to an input point. Once this is done for any surface measurement data, including errors, can be simulated as sampled on the surface and the algorithm should be able to compute a reliable fit in arbitrary precision. Thorough testing of these new algorithms is critical, and this work has already begun. The necessary input functions for a dozen shapes, prismatic and non-prismatic are being created. These functions in combination with known results, where applicable, from the Algorithm Testing Service 2.0 will be used to help ascertain the correctness and generality of these new reference fitting algorithms. The algorithms appear to work well for two geometries tested so far.

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## EARLY BALL BAR MEASURING MACHINE TESTS ENCOURAGING

NIST's Large Scale Coordinate Metrology Group is testing a newly designed ball bar measuring machine on loan from the University of Florida (UF) Machine Tool Research Center. Designed and funded as part of the Shop Floor as a National Measurement Institute Program, this machine is a single purpose measuring machine intended for determining the length of ball bars with extremely low uncertainty. Ball bars are frequently used as transfer standards of lengths, especially for Coordinate Measuring Machines (CMMs). As CMM usage becomes more widespread, the need for rapid and accurate measurement of ball bars is growing. This machine allows the length of a ball bar to be traceable to the SI unit through laser interferometers using an ingenious self-mastering technique.

UF professors consulted with NIST staff on the initial design. The instrument uses air bearings, laser interferometers, and kinematic mounts to measure ball bars. The machine will allow ball bar calibrations to be performed as accurately as the best CMMs, but much faster and at lower cost.

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## EXTENDED TEMPERATURE RANGE FOR CALIBRATION OF TWO-COLOR PYROMETERS

Properties of metallic and ceramic coatings produced by the thermal spray process depend strongly on processing conditions. Two-color pyrometry is the most common technique used to determine particle and substrate temperatures, but incorrect calibration procedures can result in significant errors. One error source of is the use of the radiance temperature rather than the absolute temperature to calibrate the pyrometer. The radiance temperature of a test surface is equivalent to the temperature of a perfectly radiating surface (e.g., blackbody,  $\varepsilon = 1$ ) with the same radiant intensity as the test surface at a specified wavelength. Tungsten ribbon lamps, the most readily available temperature standards, are usually supplied with calibrations to radiance temperature since they are normally intended for calibrating single-color pyrometers, where radiance temperature is appropriate. Another error occurs if the deviation of tungsten from gray body behavior (wavelength independent radiance) goes uncorrected. In addition to these errors, extrapolation beyond the range of the NIST calibrated tungsten ribbon lamps, will further degrade the accuracy of these temperature measurements.

NIST researchers have developed calibration procedures for two-color pyrometers that improve the accuracy of thermal spray particle temperature measurements. The errors associated with the use of radiance temperature rather than true temperature in calibrations were quantified, and the relative accuracy of determining true temperature from literature emissivity

data versus direct measurement are investigated by comparing both corrections to a blackbody calibration. Errors associated with the deviation of tungsten from gray body behavior were also investigated in this manner. In addition, the calibration range has been extended beyond that which can be achieved with lamp standards to the melting point of tungsten using facilities in the NISTs Subsecond Thermophysics Laboratory. More accurate particle temperature data will lead to an improved ability to predict coating characteristics from spray processing conditions.

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## NIST INSTRUMENTATION INSTALLED AT PLASTICS FILM MANUFACTURING FACILITY

Instrumentation developed at NIST has been installed in a polymer processing plant to facilitate development of manufacturing conditions for polymer films. The instrumentation addresses the need of polymer film producers for a more rapid measurement of molecular orientation and temperature during film production to avoid costly time delays and rejected product runs. The performance properties of biaxially stretched films are determined by their molecular orientation (or anisotropy) and the temperature at which they are processed. Currently, film processors measure orientation in post processing quality control experiments that are carried out with a considerable time delay after processing.

Under an arrangement with a private company, NIST scientists have developed a sensor and measurement system for real-time monitoring of temperature and molecular orientation during processing of biaxially stretched polypropylene film. At the private company facility, film processing is carried out in a continuous manner using a tenter stretching frame in an oven that stretches a plastic ribbon in two perpendicular directions: the process flow direction (machine direction) and the direction perpendicular to the process flow (transverse direction).

The NIST measuring technique relies on the presence of a fluorescent dye molecule that is doped into the polymer resin at very low concentrations. Temperature monitoring is carried out by measuring temperature induced changes in the shape of the fluorescence spectrum. Molecular orientation is determined by measuring fluorescence anisotropy of the fluorescent probe molecule whose orientation mimics the orienta-

tion of the resin matrix. A sensor head containing optical fibers, focusing lenses, and polarization elements is lowered into the stretching oven and is positioned directly above the processed film. The optical fibers transmit light from the light source to the film and collect fluorescence light for transmission to the detectors. As the film is transported past the sensor, three quantities are measured: orientation in the machine direction, orientation in the transverse direction, and temperature. Private company scientists are using this new information to develop processing strategies that are designed to tailor product performance to specific applications.

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## AFM AT NIST INVESTIGATES NANOSCALE ELASTIC PROPERTIES

The ever-decreasing length scales in many fields of technology require new non-destructive measurement tools that can cope with submicrometer dimensions. Specifically, the ability to determine mechanical properties on the nanoscale is needed in many applications, particularly in microelectronics. To meet these needs, NIST is developing measurement tools that exploit the spatial resolution of atomic force microscopy (≈10 nm to 100 nm). The NIST approach, called atomic force acoustic microscopy (AFAM), involves vibrating the cantilever at ultrasonic frequencies to excite mechanical resonances. By measuring the resonant frequencies under both free space and surface-coupled conditions, quantitative information about the sample's elastic properties can be extracted.

With AFAM, a value of 67 GPa $\pm 7$  GPa was obtained for Young's modulus of a 1 mm aluminum film. This compares favorably with literature values of 67 GPa to 71 GPa for bulk aluminum, and a value of 68.6 GPa $\pm 0.2$  GPa obtained on the same film using surface acoustic wave methods. Furthermore, by holding the excitation frequency constant and measuring the cantilevers vibration amplitude as the tip is scanned across the sample, qualitative images can be created. NIST has obtained preliminary elasticity images of a damascene copper/SiO<sub>2</sub> dielectric test structure for microelectronics. Such images of relative elasticity may provide valuable information about elastic stiffness variations from one sample region to another.

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#### NIST STARTING DMATS PROGRAM

NIST is conducting a program aimed at reducing the laboratory-to-laboratory variance in the performance specification and characterization of electronic displays. By advancing new measurement methods and supporting standardization in the colorimetry and photometry of displays, the project will enable higher accuracy color reproduction and thereby will enhance electronic commerce.

A key element of the project is the design and construction of a standard reference light source, the Display Measurement Assessment Transfer Standard (DMATS, pronounced dee-mats). The DMATS optical targets strategically sample the entire color gamut and luminance range encountered in electronic imaging. As importantly, however, the DMATS is designed so that the arrangement of the various measurement targets present to the metrologist the sorts of stray light contamination and alignment problems likely to contribute to display measurement error. Comparison of various measurements in relation to NIST measurements will indicate measurement difficulties. Suites of targets are included to evaluate linearity of measurement instruments. Other targets diagnose uncompensated polarization effects occurring in instrument optics, ambient illumination in the measurement environment. and instrument calibration errors.

Once assembled and characterized, the DMATS units will be circulated among participating display measurement laboratories in a star intercomparison. Analysis of the data collected in the initial intercomparison will provide the first large-scale baseline assessment of display measurement uncertainties. This assessment phase will provide both motivation for and diagnostic information to support industry standardation of display measurement protocols. In addition, it will enable improvements in uncertainty estimation for various types of display measurements and for various classes of light measuring instruments. The DMATS team will disseminate measurement methods and data via a web page, and consult with industry groups and individual laboratories to foster standardization of measurement practices. A second intercomparison will be conducted with laboratories using standard measurement procedures, many of which were developed at NIST, in order to evaluate measurement improvements and any consequent reduction uncertainties.

Thereafter, DMATS units, possibly commercially produced and calibrated by NIST, can be made available to laboratories for periodic self-certification of display measurement environments, instruments, and procedures.

The DMATS initiative was received enthusiastically by industry representatives at recent technical conferences sponsored by the Society for Imaging Science and Technology, the International Society for Optical Engineering, the International Commission for Illumination, the Council for Optical Radiation Measurements, and the Society for Information Display. CONTACTS: Kevin Brady, (301) 975-3644; kevin. brady@nist.gov and John Libert, (301) 975-3828; john.libert@nist.gov.

#### NIST LEADS INDUSTRY ROUND-ROBIN OF PHOTONIC SEMICONDUCTOR CHARACTERIZATION

NIST is conducting an industry interlaboratory comparison of *ex situ* characterization of InGaAsP films on InP that are the basis of long-haul telecommunication components. Lack of standardized assessment procedures for InGaAsP was identified as a major impediment for photonics manufacturers at the Optoelectronics Industry Development Association Metrology Workshop.

The characterization techniques under study are x-ray rocking curves, photoluminescence, and photoreflectance. The x-ray technique and photoluminescence are commonly used to qualify wafers for device processing. Wafers having compositions corresponding to emission wavelengths ranging from 1.0 mm to 1.55 mm were obtained from industry. Mapping of full wafers demonstrated that uniformity is clearly an issue in this material system, therefore the specimens in the study are from the most uniform part of each wafer. Participants in the round-robin include laser manufacturers, epitaxial film suppliers, and measurement instrumentation manufacturers. The first participant, a major optoelectronics manufacturer, stated that their involvement had already been very valuable; the process of measuring the specimens uncovered assumptions that had limited their understanding of their data and its accuracy.

NIST's development of accurate assessment procedures for InGaAsP will enable sharing of data between laboratories and improve predictive modeling for manufacturers of semiconductor lasers, detectors, and amplifiers. This is especially critical in light of the increasing industry trend to outsource the growth of epitaxial layers.

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#### NEW NIST MEASUREMENTS ADDRESS RESPONSE OF MEDIA TO RAPIDLY CHANGING MAGNETIC FIELDS AS ENCOUNTERED IN MAGNETIC RECORDING

NIST scientists have made quantitative, vectorial measurements of magnetization dynamics of Ni-Fe magnetic thin films, simultaneously at the surface and approximately 50 nm in the interior. The measurements were performed using the linear and non-linear magneto-optical Kerr effects. These measurements address the problem of the inhomogeneous magnetization response of magnetic materials when subjected to rapidly changing magnetic fields.

When the rate of change of the applied field approaches the characteristic response time of the material (the precessional frequency, typically several gigahertz), the magnetizations response can become complicated. At these frequencies, the magnetization does not simply align with the field, but instead swings toward the field and oscillates (precesses) around it before finally settling into the field direction. However, it was not known whether the surface and the interior of the magnetic material reacted to the applied field in the same way. The linear effect is sensitive to the magnetization in the interior of the film, while the non-linear effect is sensitive only to the magnetization of the first few atomic layers.

NIST scientists induced rapid, near 90° rotations of the magnetization of Ni-Fe films in a geometry similar to that of the ferromagnetic cores of magnetic recording write heads. The rotation of the magnetization vector was probed with a 50 fs laser pulse. The system had an overall temporal sensitivity of 50 ps and a sensitivity to magnetization angle of about 3°. They found that, contrary to the expectations of some models for magnetic response, the surface and the interior region responded identically. Detailed measurements showed that the magnetization exhibited a fast rotational response over about one nanosecond followed by a smaller, slow response over tens of nanoseconds.

These effects will soon be important in magnetic data storage devices as their data rate increases. Disk drives store information by switching small regions of magnetic material to represent binary data. It is necessary to have a detailed understanding of the induced precessional magnetodynamics in order to optimize the recording process. Modeling of these large, rapid magnetization motions in real materials is difficult, so direct dynamic measurements are important for continued device development.

Work is ongoing to study the dependence of the response on the thickness of the film to find when the response of the surface might deviate markedly from the interior due to eddy currents. The results were presented at the Magnetic Recording Conference in Minneapolis in August 2001.

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## SOMETHING NEW UNDER THE SUN: ENERGY WALLS

Lean against a buildings wall on a hot summer day and you can feel the suns reflective heat. To better tap this under-used energy resource, builders and renewable energy experts are integrating electricity-producing photovoltaic (PV) cells into the exterior building envelope. Increasingly, PV modules are being considered as replacements for conventional facades, roof tiles, asphalt shingles or even shading devices.

Based on guidance received from leading manufacturers of PV cells, NIST has launched a multiyear research program to provide needed performance data on this innovative technology. The project involves a mobile tracking PV test facility, a meteorological station and a building-integrated PV testbed. The mobile facility records the effects of the angle, temperature and solar spectrum on the electrical performance of various PV panels. The meteorological station measures solar radiation, wind and temperature conditions during the performance monitoring of building-integrated PV panels. The testbed conducts side-by-side comparisons of four different types of PV panels-crystalline, polycrystalline, silicon film and amorphous silicon. Each PV technology is represented in the testbed by two panels: one without any thermal insulation behind it (representative of window and skylight applications) and one insulated to simulate opaque wall applications. The energy produced by each of these eight panels and the coincident meteorological conditions are recorded every five minutes for a year.

NIST researchers are using the data to improve computer simulation tools that predict the performance of building-integrated PVs for various geographic locations and building orientations. The payoff for success could be considerable, since residential and commercial buildings utilize more than 40 % of the electricity consumed in the United States.

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## U.S. ECONOMY REAPS BENEFITS OF JOSEPHSON VOLT STANDARD

Sometimes its hard to quantify a scientific advance, but in the case of a standard developed to accurately measure the volt, impact can be assessed. In fact, a recent economic study commissioned by NIST finds that the standard enjoys a 5-to-1 benefit-to-cost ratio.

In the late 1960s, the relationship between voltage and frequency was measured precisely with newly discovered superconductor devices called Josephson junctions. Over the years, NIST developed two standards for the volt based on Josephson technology—the 1 V and 10 V. The 10 V technology was the version most in demand by industry and other government agencies for use in calibration facilities.

After developing the technology, NIST transferred it to a private company which began marketing Josephson array systems in 1996. Today, there are at least 16 of these systems in operation in the United States.

A basic standard, such as that enabling high-accuracy voltage measurement, is at the apex of an industry supply chain and can, therefore, have economic impact on a number of industries. In this case, the Josephson volt standard infratechnology transferred to industry by NIST is used in the development and marketing of high-accuracy solid-state voltage sources and associated calibration services. These instruments and services are purchased by the measuring instrument industry to manufacture precision voltage measuring devices, such as digital voltmeters. Finally, the productivity of companies that use these devices, such as those in the aerospace industry, is increased.

In order to derive a benefit-to-cost ratio, economists compared the constant dollar benefits versus the constant dollar costs from 1987 (when the first systems became available) through 1999. The ratio, they found, was 5-to-1, and the net benefit was estimated at \$45 million.

The report, Economic Impact Assessment of the NISTs Josephson Volt Standard Program, is available at www.nist.gov/director/planning/strategicplanning.htm or by e-mail at gregory.tassey@nist.gov.

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#### **BALDRIGE CRITERIA: GOOD TO GO FOR 2002**

Is your organization thinking about applying for the 2002 Malcolm Baldrige National Quality Award? If so, you can start now, basing your application on the 2001 Baldrige Criteria for Performance Excellence.

The criteria—which are reviewed annually by NIST and many outside experts—underwent significant changes in 2001. These included highlighting the increasing importance of e-commerce, the use of Internet-based interactions and the importance of aligning all aspects of a performance management system. To give organizations more time to better understand and implement these changes, the awards board of private-sector overseers has recommended that the 2001 criteria not be revised.

Over the years, the criteria have focused more sharply on overall performance excellence and results. In addition to being the basis for a Baldrige Award application, thousands of organizations use the criteria to assess and improve performance.

Single copies of the 2001 Baldrige Criteria for Performance Excellence are available free of charge from NIST by calling (301) 975-2036, faxing a request to (301) 948-3716 or sending e-mail to nqp@nist.gov. They also may be downloaded from www.quality.nist.gov. Packets of 10 or more, as well as other Baldrige-related materials, may be ordered for a fee from the American Society for Quality, (800) 248-1946, www.asq.org.

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## NIST ASSESSES ACCURACY OF THIN FILM DIMENSIONS

The thickness of gate dielectricsthe ultra-thin insulating films that separate electrical gates and channels in transistorsis the smallest dimension to be measured on a chip. As thickness requirements fall below 4 nm, films must be produced with a thickness tolerance of less than 0.3 nm, and metrology tools with a precision of better than 0.1 nm are required. NIST is developing a new data analysis approach that quantifies the errors associated with these thickness measurements, a critical issue in semiconductor manufacturing and performance.

Using high-resolution transmission electron microscopy to measure thickness, new methods have been developed for obtaining two-dimensional calibration information from an image of the silicon lattice substrate. The ruler is the distance between silicon atoms, measured in a new way that significantly improves measurements of atom position. The approach involves computer-based image processing and a mathematical algorithm. The image of the lattice is digitized, a computer is used to define where the atoms are, and the algorithm produces a measurement based on multiple inputs.

Films nominally 2 nm thick were measured with an estimated uncertainty of 0.2 nm, confirming that standard techniques are not good enough. The researchers now are working on improving the measurement precision and understanding accuracy issues. The accuracy is estimated by computer modeling of gate dielectric stacks using simulation software. NIST researchers are quantifying the effects of variables such as microscope lens aberrations, vibration and sample properties on the accuracy of the thickness measurement and are searching for the optimal imaging conditions.

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## NEUTRON PROBES FOCUS THE SEARCH FOR INSULATORS

The semiconductor industry is desperately seeking better insulating materials. By the end of the decade, state-of-the-art integrated circuits will contain more than a billion transistors, connected by thousands of meters of ultra-fine copper wiring. Without satisfactory replacements for silicon dioxide, the current insulator of choice, the potential for electrical cross talk between such closely packed wires will be enormous.

Recently, the search has become less frenzied and more focused, thanks to a productive collaboration between NIST and International SEMATECH, the consortium of chip makers and equipment suppliers. The team has pioneered the use of powerful analytical methods to gather critical information on the properties of a promising and extremely varied class of insulators: thin films with nanometer-scale pores.

The pore structure lowers the so-called dielectric constant of the films to desired levels. Unfortunately, the Swiss-cheese-like effect can undermine other important properties—from mechanical strength to hardness to thermal expansion—that strongly influence the suitability of the films for semiconductor manufacturing.

Working at the NIST Center for Neutron Research, the team is using small-angle neutron scattering to characterize a growing cast of nanoporous, low-k dielectric films. With the tools, researchers can measure average pore size, pore distribution and connectivity, film thickness and composition, coefficient of thermal expansion, and other important characteristics of samples.

NIST has evaluated an assortment of nanoporous films supplied by International SEMATECH. These include spun-on glass materials, films from chemical

vapor deposition and organic thin films. Structural information provided by NIST is placed into a master database, where it is combined with other data needed to evaluate candidate materials. Materials and equipment suppliers also are leveraging the NIST-provided measurements to engineer novel nanoporous films with even more complex microstructures, broadening the search for economical, high-performance low-k dielectric films.

An overview of this project and others in the Interconnect and Packaging Metrology Program can be found at www.eeel.nist.gov/omp/interconnect.html. For technical information, contact Barry Bauer, (301) 975-6849, barry.bauer@nist.gov, Eric K. Lin, (301) 975-6743, eric.lin@nist.gov or Wen-li Wu, (301) 975-6839, wen-li.wu@nist.gov.

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## TEMPERATURE MEASUREMENT IN MICROFLUIDIC DEVICES

NIST scientists have developed a new method for measuring the temperature in microfluidic devices. Microfluidic, or so-called "lab-on-a-chip," devices are miniaturized chemical and biochemical analysis systems that may one day replace conventional bench top instruments. The ability to measure and control fluid temperatures within lab-on-a-chip devices can be very important for efficient electrophoretic separations and for enzyme-activated reactions. In spite of its importance, only a few techniques have been reported for the measurement of fluid temperatures in these hair-thin fluid channels. The drawbacks to these techniques are that they are difficult to implement (involving either NMR or Raman spectroscopy) and are limited in resolution, so that rapid spatial and temporal variations of temperature cannot be measured. The new temperature measurement technique developed at NIST is simple to implement, requiring only standard video and fluorescence microscopy equipment, and can be used for temperature measurements with simultaneous micrometer spatial resolution and millisecond time resolution. The measurement technique is based on the temperature dependence of the fluorescence intensity of a dilute fluorophore added to the fluid, and thereby takes advantage of the inherently high signal-to-noise ratio of fluorescence-based measurements. A description of the technique was recently published in the journal Analytical Chemistry. CONTACT: David Ross, (301) 975-2525; david. ross@nist.gov or Laurie Locascio, (301) 975-3130; laurie.locascio@nist.gov.

#### HIGH TEMPERATURE LEAD-FREE SOLDERS

Several industries have identified needs for solders that perform reliably at ever-higher temperatures, temperatures which approach the melting point of the industry-standard tin-lead eutectic alloy ( $T_{\rm m} = 183$  °C). For instance, automotive under-the-hood microelectronics are subject to higher ambient temperatures due to low hood profiles (less air circulation) and the need to place assemblies, such as engine control modules, near the point of application to reduce wiring harnesses. Such harsh environment applications require solders with melting points higher than that of tin-lead eutectic in order to achieve the required reliability. In addition, recent legislative and marketing pressures in Europe and Japan have pushed U.S. manufacturers to pursue lead-free solders for all microelectronics applications, leading to the double challenge of developing high temperature, Pb-free solders.

This year the National Center for Manufacturing Sciences (NCMS) completed the 4 year High Temperature Fatigue Resistant Solder Project with the assistance of NIST. The goal of the project was to determine whether highly reliable, non-toxic, cost-effective substitutes could be found for industry-standard high lead and Sn-Ag eutectic solders in harsh environments.

NIST played a leadership role in this research project, chairing the Alloy Task Group and providing definitive, timely phase diagram data and critical analyses needed for evaluating candidate solders. NIST also coordinated the final analysis of the data, the determination of conclusions and recommendations, and the writing of the NCMS final report. Six lead-free solder compositions were identified that exhibit fatigue performance during cycling up to 160 °C of a wide range of surface mount components which is superior to the industry standard eutectic Sn-Ag alloy. In addition, seven lead-free alloys cycled with ball grid array packages through 40 °C to 125 °C strongly outperforming Sn-Pb eutectic solder.

The High Temperature Fatigue Resistant Solder Project was summarized in an invited article in *Advanced Materials and Processes*, April 2001, written by NIST staff. In addition, a technical paper presented by NIST was selected as one of the five best papers out of 58 presented at the TMS 2001 Annual Meeting Symposium on Recent Progress in Pb-Free Solders and Soldering Technologies.

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#### MOLECULAR DYNAMICS SIMULATIONS AND NEUTRON SCATTERING YIELD NEW INSIGHTS INTO PROTEIN DYNAMICS

Understanding the process by which a protein efficiently folds to the physiologically active native state is one of the great challenges in biology. Proteins can also form stable, partially folded states that are thought to resemble intermediate states along the protein folding pathway. Recently, a collaboration between the University of Pennsylvania, the University of California at Irvine, and NIST have used molecular dynamics simulations and quasielastic neutron scattering techniques to elucidate dynamic changes in the protein alphalactalbumin for the native state and the partially folded molten globule state.

Molecular dynamics simulations are the ideal complement to neutron scattering because neutrons measure correlation functions that are based on the positions of the atoms as a function of time. For the case of alpha-lactalbumin, the neutron results show that the side-chain protons in the molten globules are significantly more mobile than those in the native protein. Molecular dynamics results, which reproduce the measured quasielastic neutron spectra extremely well, show that the observed dynamic changes arise primarily from the particular region of the protein that forms a beta sheet in the native state and unfolds to a random coil in the molten globule. Thus the spectroscopic results clearly reflect the formation of secondary structures that occur as a protein folds. Moreover, the techniques developed for the comparison of molecular dynamics simulations with neutron spectra are directly applicable to a wide variety of complex materials.

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#### VIDEO MEASUREMENTS WITH M48 COORDINATE MEASURING MACHINE

The NIST high accuracy M48 coordinate measuring machine has been retrofitted with a video microscope system to calibrate two-dimensional grid plates. Preliminary measurements on a 200 mm by 200 mm grid show a reproducibility of 0.050  $\mu m$  (1  $\sigma$ ) for measurements in multiple orientations. Based on these measurements, the instrument should be able to produce calibrations of grids up to 600 mm square with an uncertainty of 0.2  $\mu m$  or better.

This uncertainty is somewhat better than has been achieved by the M48 when measuring other two-dimensional artifacts (ball plates and hole plates), primarily because of the superior repeatability of the video probe system relative to the mechanical contact probe normally employed by the M48. There are indications that the accuracy may be further improved by correcting for machine straightness errors, which are on the order of 0.1  $\mu$ m or less. These errors were previously considered to be too small to be significant, but with the video probe system even an error as small as 0.050  $\mu$ m now can be detected easily and is worth correcting.

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# INTEROPERABILITY TESTING TOOLS FOR METROLOGY EQUIPMENT SUCCESSFULLY DEMONSTRATED

Shortening time to market is a strategic imperative for U.S. manufacturers in an increasingly competitive global marketplace. Achieving a seamless flow of data from computer aided design part models to equipment on the factory floor is a key requirement to shorten product cycles. In the area of metrology equipment, one of the key interfaces that NIST has identified the interface between the metrology hardware, the coordinate measuring machine, for example, and the control system for that hardware. NIST is working with the Metrology Automation Association to develop a standard for this interface, which is called the Common Machine Interface (CMI).

Industry is setting the specifications and NIST is providing testing tools. In July, NIST researchers teamed with one of the leading manufacturers of metrology equipment, to demonstrate the CMI test suite. Demonstrated were the NIST-developed common sender and receiver utilities, a common test artifact, and an implementation of CMI on a coordinate measurement machine. The demonstration of the test suite included the transmission of a CMI compliant test file from the NIST lab to the manufacturers' facility in the U.K., execution of the file on the coordinate measuring machine to inspect features on the test part, monitoring and collection of status information, and review of test log files with preliminary analysis tools.

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## NIST REDUCES UNCERTAINTY FOR NIST CAPACITANCE CALIBRATIONS

Recent work at NIST has resulted in a factor of three decrease in the uncertainty for certain of the highest-

level calibrations of fused-silica capacitance standards. At NIST, capacitance calibrations are based on the SI farad as realized by the NIST Calculable Capacitor. Until recently, measurements on the Calculable Capacitor had been performed only at a specific frequency, 1592 Hz. As a result, the uncertainty budget for NIST capacitance calibrations included a component to account for the uncharacterized frequency dependence of NIST standards between 1592 Hz and the frequency at which calibration measurements were performed (100 Hz, 400 Hz, or 1 kHz, for fused-silica capacitance standards). NIST staff have recently completed a detailed characterization of the transfer of the farad unit from the Calculable Capacitor to Calibration Laboratory reference standards at a frequency of 1 kHz. As a result, the relative uncertainty (95 % confidence) for the calibration of 10 pF and 100 pF fused-silica capacitance standards has decreased from  $1.5 \times 10^{-6}$  to  $0.5 \times 10^{-6}$ . Stenbakken, CONTACT: Gerard (301) 975-2440; gerard.stenbakken@nist.gov.

#### NIST SPEARHEADS GUIDE FOR FIRST RESPONDERS ON COPING WITH ELECTRONIC EVIDENCE

Meeting strongly expressed needs of the criminal justice community, the NIST Office of Law Enforcement Standards (OLES) has completed a guide on electronic evidence for law-enforcement personnel first responding to a crime scene, a challenging and increasingly important new forensic area. The guide has been published by the National Institute of Justice as Electronic Crime Scene Investigation: A Guide for First Responders. OLES led the activity resulting in the guide through identification of and contact with national and international forensic professionals experienced in the details of handling electronic crimes. OLES was able to bring together a disparate group of people and through various interactions, including meetings at NIST, was able to develop a series of consensus positions for publication.

Law enforcement officers called out to investigate a complaint or criminal activity in progress will have this added resource available, the first in a series devoted to electronic evidence and associated issues. The guide offers simple explanations on how to recognize potential electronic evidence, how to collect and transport the items or media in a fashion that will maintain the integrity of the evidence and meet challenges in the courtroom. The guide can be downloaded in ASCII text and .pdf format from the NIJ web site; http://www.ojp.usdoj.gov/nij/pubs.htm, using the locator number NCJ 187736.

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# NEW SOURCE OF SUPERCONDUCTOR MEASUREMENT MISINTERPRETATION DISCOVERED

As part of NIST's program to develop standard measurement techniques for superconductors, two NIST scientists have identified and studied a new source of misinterpretation in critical current measurements of superconductors. The critical current is the maximum current a conductor can carry before a quench, when it reverts to the normal resistive state. Researchers can tell when the critical current is reached by measuring the resistive voltage on pairs of voltage taps soldered to the superconductor wires.

However, the NIST scientists discovered that anomalous inductive voltages can be induced in the loop formed by the voltage taps. The inductive voltages vary systematically with current, current sweep direction (increasing or decreasing), applied magnetic field, and whether the specimen was driven into the normal state in an immediately preceding measurement. Furthermore, the decay time of the inductive voltage signal after ending the current ramp is longer near the onset of the resistive transition. These decay times are even longer during a superconductors first current sweep after a quench.

Many superconductor applications now require higher current densities, larger wire diameters, and less copper stabilizer, all of which result in marginally stable conductors with high critical currents above 1000 A. Variable induced voltages and long decay times become a concern when currents or current ramp rates are high or when voltage curves need to be extrapolated for measurements on marginally stable conductors. The resulting data can be mistakenly attributed to a bad conductor, a damaged specimen, an electrical ground loop, a low critical current, or specimen motion in the background magnetic field.

To avoid anomalous induced voltages, the NIST scientists recommend cycling the current before acquiring data after a quench, avoiding data acquisition while the current is being ramped, and allowing 3 seconds of settling time after current levels are changed before measurements are made near the critical current.

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#### QUANTIZED VOLTAGE STEPS IN ARRAYS OF STACKED JOSEPHSON JUNCTIONS DEMONSTRATED

NIST researchers have measured the first constant voltage steps in stacked Ti-barrier Josephson junctions. This is a significant milestone toward creating lumped arrays of Josephson junctions for ac voltage standards.

For these standards many junctions must be tightly packed into a size smaller than a quarter wavelength of the microwave drive frequency. This ensures that all the junctions see nearly the same microwave power. For ac and dc programmable voltage standards, lumped arrays will enable increased output voltage, increased bandwidth, increased operating margins, and lower power operation.

One thousand double-junction stacks, for a total of 2000 series-connected junctions, were fabricated. The 40 nm titanium barriers are separated by only 50 nm of niobium. When a 16 GHz microwave bias was applied, a constant voltage step was measured at the expected quantized value of 66.17 mV. The voltage of the step was constant over a 3.5 mA current range which is more than sufficient for operation as a programmable voltage standard array. This observation of constant voltage steps demonstrates that the stacked junctions are sufficiently uniform for application in voltage standard circuits. For each increase in the number of junctions per stack, an improvement in operating margins is expected. Adding a third and fourth junction to each stack will be the next step toward the goal of fabricating and observing steps in 50-junction stacks in order to create a 2 mm long lumped array with 50  $\Omega$  impedance.

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# SUPERCONDUCTING DETECTOR ARRAY SUCCESSFULLY DEPLOYED THROUGH NIST COLLABORATION

In collaboration with the NASA/Goddard Space Flight Center on June 2, 2001, NIST successfully deployed a new type of superconducting detector at the Caltech Submillimeter Observatory at the summit of Mauna Kea, Hawaii. The FIBRE instrument consists of a Fabry-Perot spectrometer with an 8 pixel detector array of superconducting transition-edge sensor (TES) bolometers. The array is read out using a superconducting quantum interference device (SQUID) multiplexer. Both the bolometer array and the SQUID multiplexers were invented and fabricated at NIST. This technology is a revolutionary breakthrough for materials analysis, astronomical and other photon detection applications. FIBRE is the first astronomical demonstration of both TES submillimeter bolometers and SQUID multiplexers. Although the weather was extremely poor, the spectrometer detected the limb of the moon with a spectral resolving power of ≈1100 at a wavelength of 365 µm. In better weather conditions, FIBRE will be used to study CO rotational lines and the fine structure line of Cl in distant galaxies.

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## NIST OBTAINS MICROWAVE IMAGES USING ITS NEAR-FIELD RANGE FACILITY

NIST has processed spherical scanning data to obtain a microwave image of a bicycle and a ladder using its spherical near-field measurement facility. The technique was originally conceived in a NIST theoretical paper, and was implemented on the NIST spherical near-field scanning facility in Boulder. The objective is to develop a method for the precise evaluation of incident electromagnetic fields in the quiet (measurement) zones of anechoic chambers, and far-field and compact antenna measurement ranges. The microwave image was obtained at 16 GHz and compared to an optical image. The microwave image of the bicycle shows considerable structural detail. For example, the wheels, saddle, fork, and frame are visible. While this image was focused on the bicycle, the horn antenna that is the source of the microwave signals and a step ladder are out of focus but visible in the microwave picture. CONTACTS: Andy Repjar, (303) 497-5703; repjar@ boulder.nist.gov.

#### NIST RESEARCHERS DEVELOP AND DISTRIBUTE CRUCIAL OPTICAL DISPERSION MODEL TO THE SEMICONDUCTOR INDUSTRY

NIST researchers have recently derived a unique Generalized Tauc-Lorentz (GTL) dispersion model that can successfully model spectroscopic ellipsometry data for high-k thin films in the spectral range of 1.5 eV to 6.3 eV. This model can be used to determine accurately film thickness and optical properties. Furthermore, the model and measurements were shown to provide a nondestructive method to assess film qualities such as surface roughness and degree of crystallinity. This GTL model has been implemented in a user-friendly windows-based software which was recently transferred to International SEMATECH for further dissemination to the industry. The spectroscopic ellipsometry measurements enabled by this work will provide crucial data for identifying, developing, and ultimately manufacturing high-k gate dielectrics.

One of the most important parts of the search for a new gate dielectric is the ability to measure accurately and control the film thickness. The thickness measurement method that integrated circuit (IC) manufacturers most frequently use is nondestructive, non-contact ellipsometry. However, this technique requires an optical model to characterize the film properties in order to extract an accurate film thickness. To address

this industry need, NIST researchers have been acquiring films from IC industry leaders and university research groups to investigate the optical properties and develop models required for measuring the thickness of these new materials.

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## NIST SCIENTISTS RECORD THZ SPECTRA OF BIOMOLECULES

As part of a competence-funded program in THz spectroscopy and imaging, NIST scientists have developed a novel THz spectrometer for investigating large-amplitude vibrational motions found in many biomolecules. These vibrational motions are responsible for the flexibility of biomolecules necessary for a protein to fold. The capabilities of the instrument have been demonstrated by recording THz spectra of two biological molecules: biotin and riboflavin.

THz radiation is produced in a process called "difference-frequency generation" by coupling two near-infrared laser beams fed from an optical fiber onto a GaAs photomixer. The THz radiation is directed through the sample and then detected by a liquid Hecooled bolometer. A significant effort was made to eliminate the intense atmospheric water-vapor absorption present in the THz region, and the etalon or standing-wave structure, which often limits sensitivity. To eliminate these effects the photomixer, sample, and bolometer were all placed in a common vacuum system, with no windows separating them.

The measurements were made at 4.2 K to increase spectral resolution, and the resulting spectra reveal a number of low-frequency vibrations at frequencies less than 4 THz. The spectra are unique in that they are not complicated by the presence of artifactual etalon peaks, which have plagued many previous THz studies. The fact that these low frequency vibrational modes were observed in solid samples indicates that the associated nuclear motions do not correspond to large geometry changes in the molecules. Efforts are presently under way to identify these motions with specific nuclear displacements.

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#### NIST DEMONSTRATES QUANTUM-MECHANICALLY-ENTANGLED, SPIN-SQUEEZED STATES

By applying coherent laser beams to trapped ions, NIST staff in Boulder have generated quantum-mechanicallyentangled, spin-squeezed states, and, for the first time, shown that such states can be used to increase measurement precision beyond that which is possible without the use of entanglement. The results of these studies have recently been reported in Physical Review Letters. As a demonstration, the NIST researchers produced spin-squeezed states of two beryllium atomic ions and showed that when the spins are rotated in a magnetic field, the uncertainty in determining the rotation angle is smaller than can possibly be obtained if the atoms are not entangled. Such techniques are an integral part of the emerging fields of quantum logic and quantum information, but can also be used to improve sensitivity in spectroscopy and reduce noise in atomic clocks.

Although spectroscopic precision can be improved without the need for entanglement by increasing the number of atoms observed, in atomic clocks based on ions, however, the quest for accuracy requires the use of only a small number of ions. With a potential relative uncertainty of 10<sup>-18</sup>, these techniques should find application in the development of new optical ion clocks. CONTACT: Dave Wineland, (303) 497-5286; wineland@boulder.nist.gov.

of polymeric fluids. The new standard fluid is certified by NIST for the shear-rate dependence of viscosity and first normal force difference at shear rates from 0.001 s<sup>-1</sup> to 100 s<sup>-1</sup> over a temperature range from 0 °C to 50 °C. The linear viscoelastic storage modulus and loss modulus are also certified at frequencies from 0.04 rad/s to 100 rad/s over the same temperature range. The new fluid supersedes the previous Standard

better methods of measuring the rheological properties

The new fluid supersedes the previous Standard Reference Material 1490 Non-linear Fluid Standard, which was composed of a high-molecular-mass polyisobutylene dissolved in normal hexadecane. The new standard fluid SRM 2490 consists of a similar high-molecular-mass polyisobutylene dissolved in 2,6,10, 14-tetramethylpentadecane (common name pristane). Pristane is a branched alkane of a slightly higher molecular mass than the normal hexadecane; the branching prevents crystallization or vitrification down to -60 °C, while the higher molecular mass reduces the rate of evaporation of pristane as compared to normal hexadecane.

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#### **Standard Reference Materials**

## STANDARD NON-NEWTONIAN FLUID FOR RHEOLOGICAL MEASUREMENTS

A new Standard Reference Material (SRM), SRM 2490, is available for calibration of rheometers, instruments used to measure the flow behavior of fluids. Complex fluids, such as suspensions or polymer melts and solutions, often do not follow the simple Newtonian ideal in their flow behavior. Such fluids are found in numerous applications in everyday life (injection molding, paints and coatings, food products, etc.), and the ability to measure accurately and characterize their behavior is very important to optimizing their processing conditions. Since there are a number of commonly used methods to measure the flow behavior of polymers, the new Standard Reference Material will provide a means for comparing the performance of different instruments, as well as a tool for research into

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